REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	97 JAN 24	FINAL REPORT 25 Apr 95 - 24 Oct 96

4. TITLE AND SUBTITLE

SBIR95 Doubled Diode Laser

5. FUNDING NUMBERS

65502F
3005/SS

6. AUTHOR(S)

Dr. David Deacon
Dr. Simon Field

AFOSR-TR-97
0145

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Deacon Research
2440 Embarcadero Way
Palo Alto, CA 94303

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

~~USAF, AFMC~~
Air Force Office of Scientific Research - NE
110 Duncan Avenue, Suite B115
Bolling AFB, DC 20332-0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

F49620-95-C-0037

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Distribution Statement A. Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The Doubled Diode Laser program aimed to produce a highly efficient blue laser source based on the frequency doubling of a semiconductor diode laser in a periodically poled lithium niobate waveguide. Under this program we have demonstrated waveguide doubling device conversion efficiencies of 300%/W, and generated more than 7mW of blue light. The program has enabled us to attain a unique processing capability, that of producing the required 4.5μm period domain gratings for the waveguide frequency doublers, achieved by the application of our own proprietary material processing techniques and pattering geometries.

The funding provided by this program generated key insights into the electric field poling process and the material processing required to achieve highly efficient devices. The process development started under this program has led directly and indirectly to a number of successes: highly efficient blue-frequency-doubling waveguide devices, commercial PPLN sales for infra-red devices, 3" wafer scale poling of infra-red devices, and PPLN for bulk, visible frequency conversion applications.

14. SUBJECT TERMS

DTIC QUALITY INSPECTED 4

15. NUMBER OF PAGES

7

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	

Final Report

4/25/95-10/25/96

Doubled Diode Laser

Contract Number : F49620-95-C-0037

Deacon Research
2440 Embarcadero way
Palo Alto
CA, 94303

Principle Investigator : Dr. David A.G. Deacon

Project Scientist : Dr. Simon J. Field
Tel : 415-493-6100x221

Program Manager : Dr. Howard Schlossberg, AFOSR

Executive Summary

The Doubled Diode Laser program aimed to produce a highly efficient blue laser source based on the frequency doubling of a semiconductor diode laser in a periodically poled lithium niobate waveguide. Under this program we have demonstrated waveguide doubling device conversion efficiencies of 300%/W, and generated more than 7mW of blue light. The program has enabled us to attain a unique processing capability, that of producing the required 4.5μm period domain gratings for the waveguide frequency doublers, achieved by the application of our own proprietary material processing techniques and pattering geometries.

The funding provided by this program generated key insights into the electric field poling process and the material processing required to achieve highly efficient devices. The process development started under this program has led directly and indirectly to a number of successes: highly efficient blue-frequency-doubling waveguide devices, commercial PPLN sales for infra-red devices, 3" wafer scale poling of infra-red devices, and PPLN for bulk, visible frequency conversion applications.

Introduction and Program Aims

This program has enabled a huge progression to be made in the area of electric field poling of lithium niobate for nonlinear optical devices. The aim of the program was to develop highly efficient blue laser source based on the frequency doubling of a semiconductor diode laser in a quasi-phasematched (QPM), periodically poled lithium niobate (PPLN) waveguide. The proposal was to use electric field poling to fabricate the QPM domain grating by altering the crystal structure of the $LiNbO_3$ substrate on a micron scale - a task which had never been demonstrated using commercially available (low cost) 0.5mm thick $LiNbO_3$ wafer substrates.

In the course of this program we identified a number of key insights concerning the poling process, and applied these, together with our own proprietary processing capabilities, to achieve the results described below.

1 Thermally induced processing defects

Early in this program our electric field poling efforts were plagued by randomly appearing and apparently unexplained regions where the fine (4.5µm period) features would be merged together. Using the Nomarskii differential-interference-contrast microscope purchased under this program we were able to observe domain inverted defects present in our 0.5mm thick crystal samples after photolithographic processing. but before the electric field poling process. By carefully monitoring the condition of the crystals during the lithography process we discovered that these domain defects resulted from any temperature cycling that exceeded ~90^oC, despite the fact that the Curie temperature of $LiNbO_3$ is around 1100^o C. (It should be noted that when processing 1mm thick $LiNbO_3$ such temperature induced domain inversion defects could not be produced with identical cycling parameters.) The number of defects created depended on the temperature, ramp rate and size of the sample. 3" wafers and quarter wafers become "peppered" with defects when processed with parameters which do not affect 10mm square samples.

The effect of these defects during poling at short periods was severe. Poling would nucleate within the desired masked regions, but as the domains filled out the pattern, lateral growth of bulk domains from the defect sites caused large amounts of merging between the closely spaced grating lines. This is illustrated in Figure 1, a photomicrograph of a poled grating after etching in HF acid to reveal the domain pattern. The thermal inversion defects are clearly visible between the grating regions and have caused significant merging of the shortest (4.5µm) period structure. The effect of these defects is significantly less at longer periods, where the separation of adjacent patterned domain features exceeds the size of the thermal defect domains, as can be seen from the longer period structures in figure 1 (gratings of 4.5, 9, 13.5, 18 and 22.5µm were fabricated on the same sample).

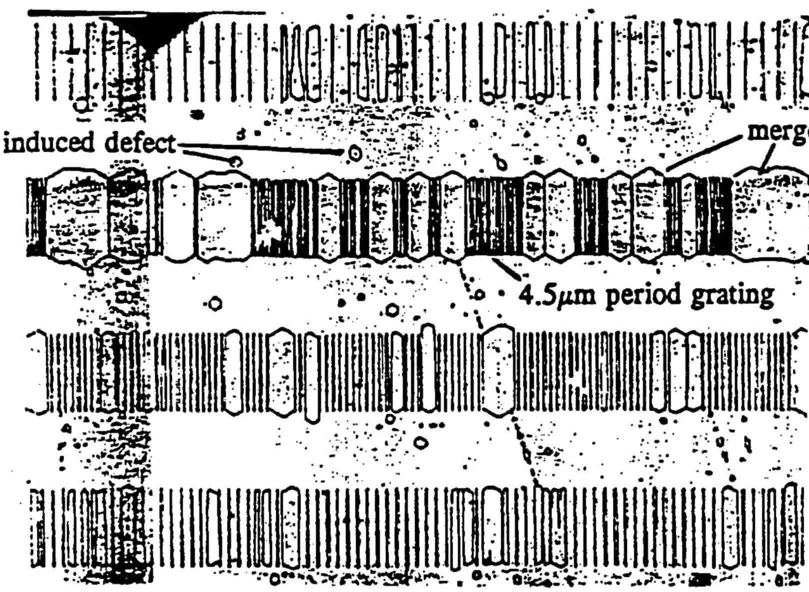

Figure 1: Photomicrograph of a poled grating sample, etched to reveal the domain pattern and the randomly distributed thermally induced domain defects.

The defects were hypothesized to result from charging of the crystal surface during temperature ramping, due to the pyroelectric nature of the $LiNbO_3$ substrate. By altering our photolithographic processing to include grounded plates to reduce surface charging we have managed to eliminate almost completely the creation of thermal domain inversion defects. Occasional isolated lines of defects are still observed on some wafers, and we believe these are related to polishing defects remaining in the wafer. The very small number of defects now present in our processed 3" wafers is low enough for us to fabricate high quality short period domain gratings, with only very occasional merged sections resulting from the influence of crystalline defects.

2 Sample Observation and Documentation

The above mentioned purchase of a 1000x Nomarskii DIC microscope also enabled us to set up a digital frame grabber system for taking photomicrographs of etched domain patterns. This capability was absolutely vital in terms of documenting the results from varied processing conditions, and in enabling us to keep track of our progress and observe the improvement in domain grating fabrication that we were able to achieve.

3 Dopants for Inhibition and Seeding

One of the major problems encountered when working on E-Field poling of $LiNbO_3$ is that of constraining the poled domains to the desired size. Simple conducting or insulating masks only provide limited control, as the domain can still expand laterally outside the desired patterned region. Of course, for long period interactions, e.g. for IR OPO's, this expansion can be allowed

for and simply designed into the mask pattern before poling. However, as the period is decreased towards that desired for QPM frequency conversion into the blue ($\leq 4.5\mu m$) the lateral domain expansion becomes comparable with the desired domain size. Thus, starting from the finite dimensions which can be lithographically produced uniformly and repeatably ($\tilde{}1\mu m$) the lateral expansion typically leads to merging between adjacent domains, or at best a non 50-50 duty cycle to the grating. Thus ways must be identified to improve the domain confinement, either by improving the mask processing, or by processing of the substrate before poling.

Our concept is that the introduction of a particular dopant species into the surface of $LiNbO_3$, may either cause seeding of, or inhibit the start of domain inversion. By appropriately patterning the doped regions and applying the high voltage pulse in the correct manner, it is hoped controlled patterned poling will be obtained, with the domain walls confined more strongly to the desired mask dimensions.

Using proton exchange to alter the chemical composition of a surface layer of the crystal we have been able to produce long period patterned poling using a uniform applied electric field. Long proton exchange times are required to drive the protons quite deep into the crystal ($\tilde{}1\mu m$) before significant effect on the poling behavior is observed. Annealing after proton exchange (to drive the protons deeper still) has also been investigated, and appears to further increase the patterning effectiveness, although the samples appear more prone to destructive electrical breakdown during poling (compared to unannealed). The protonated regions prove more resistant to domain inversion than the bulk crystal, as can be seen from the etched domain pattern of figure 2, indicating that the exchange effectively increases the coercive field required for domain inversion. The long, thin structures in figure 2 are regions of the crystal which were proton exchanged and which have not been domain inverted by the high voltage pulse. This view is of the -z face, and the proton exchange was performed through a $29.3\mu m$ period mask on the +z face of the crystal. Unfortunately however the poling inhibition effect appears too small to reliably produce short period ($4.5\mu m$) poling.

We also investigated the effects of dopant implantation from high energy ion beams. The implantation is performed through a patterned SiO_2 mask, which prevents the entry of the ions into certain regions of the crystal. Initial experiments have involved the implantation of Mg and Zn (chosen for their reported reduction of the photorefractive effect) at relatively low energies of 200keV. This gives only a very shallow penetration depth of the order of $0.2\mu m$. Poling of these samples using a uniform applied electric field indicates a weak poling inhibition or small domain wall lateral velocity decrease within the implanted region as the domain wall seems to lag slightly behind that observed in the bulk. This is shown in figure 3, where the horizontal bar indicates the ion implanted region (this etches very rapidly in HF), which was defined by implantation through a patterned SiO_2 mask. The vertical line is the domain wall, which is advancing from right to left across the figure, and which can be seen to be lagging behind in the implanted region compared to the bulk crystal.

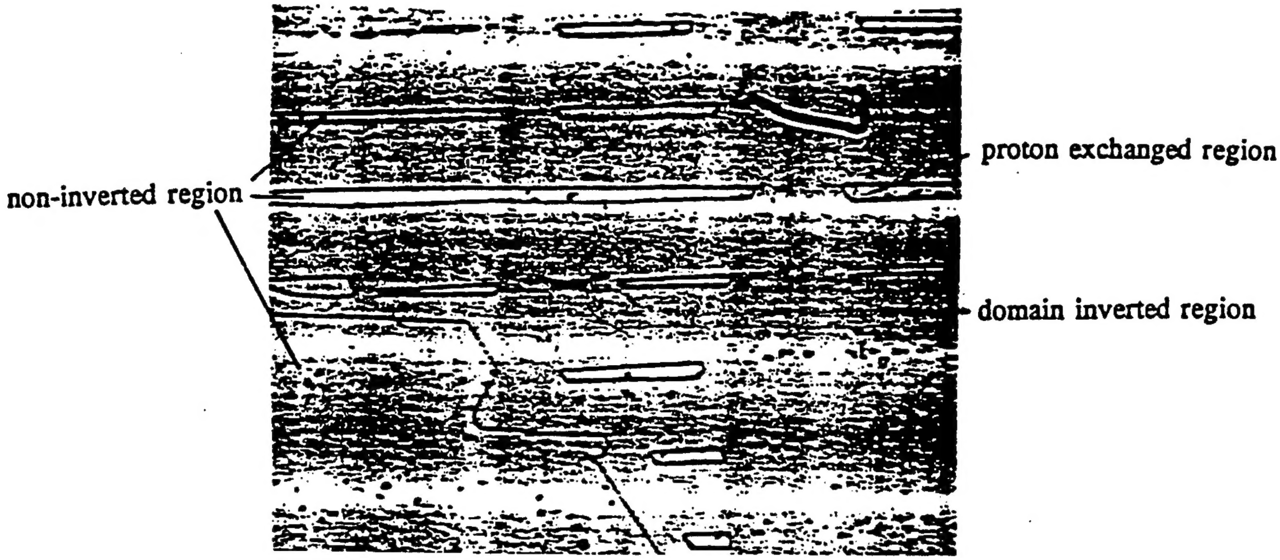

Figure 2: Etched domain pattern resulting from poling of a patterned proton exchange sample

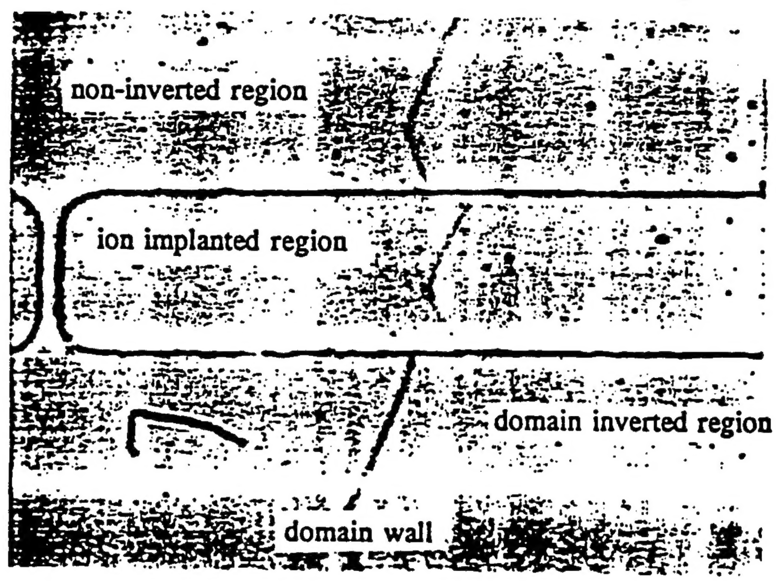

Figure 3: Etched domain pattern resulting from poling of a patterned ion implanted (Mg) sample

In view of the small apparent effect of the exchange and implantation processes on the electric field poling process, and our subsequent improved domain gratings resulting from other processing changes (described below) we did not pursue dopant incorporation any further. It may however play a role in our future processing as we try to reach steadily shorter periods and need to combine techniques to achieve the optimum result.

4 Poling of 4.5μm Period Domain Gratings

During the course of this program we made tremendous progress in the poling of short period QPM domain gratings. From our initial attempts which were dogged by merging caused by crystal defects, illustrated in Figure 1, we improved and refined our masking and processing techniques. By the end of the contract our domain control has improved to the point where we are able to fabricate 50-50 duty cycle domain gratings for waveguide frequency doubling applications, with very few merged regions or missing domains. An example of the last generation of material produced under this program is shown in figure 4, compared with the previous generation, which is the subject of the frequency doubling device reports detailed below. In part this enhanced grating fabrication was enabled by the purchase of improved diagnostic equipment under this program. A Lecroy digital storage oscilloscope with mathematical functions enabled us to record and display the poling voltage and current pulses, and to compute the area of the crystal which had actually been repoled. Comparison of poling pulse traces and the quality of the poled domain gratings enabled us to learn to recognize a well poled sample without having to etch the domain pattern.

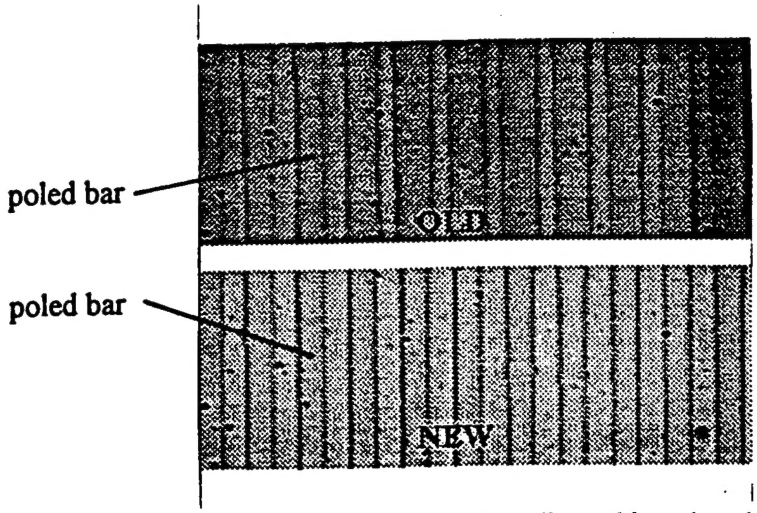

Figure 4 : Recent improvement in domain grating quality achieved under this program

5 Expansion of 4.5μm Period Poled Area

With our improved domain control capability in hand the next logical step was to increase the area of the poled sample, to enable more waveguide devices to be fabricated from a single poled chip. during the program we were successful in increasing the sample poled area to a 23mm diameter circle, the largest that would fit on a quarter of a 3" wafer and still leave room for the electrode structure to avoid breakdown around the edge of the crystal. The uniformity of the domain gratings on these larger samples was in general limited only by the quality of the phototlithography used to define the grating mask. By fabricating 23mm diameter poled areas we are now able to produce finished waveguide devices with lengths of 20mm, the performance of which is being tested under ongoing programs.

Although we did not actually perform poling of full 3" wafers under this program we did design and construct a wafer scale poling apparatus, which has since been used to successfully produce longer period poled wafers for infra-red devices. We will shortly be using this apparatus for wafer scale poling of short period domain gratings for visible frequency conversion applications.

6 Fabrication of Annealed Proton Exchanged Waveguides

In order to make an efficient frequency doubling device based on a low cost semiconductor diode laser a waveguide structure must be used to confine the pump and second harmonic beams as they pass through the nonlinear optical crystal. The waveguide structure allows much higher intensity$\times$length products than are possible with diverging gaussian beams, and the small mode size of the waveguide creates a high peak intensity for a low power. The combination of these two features enables the high conversion efficiencies required to generate mW levels of blue light.

In $LiNbO_3$ the waveguide fabrication technique of choice for frequency doubling is Annealed Proton Exchange (APE). APE waveguides can exhibit low loss, support small mode sizes, and are resistant to photorefractive damage, which is essential when generating visible light. An APE waveguide is made by immersing the $LiNbO_3$ crystal in a bath of molten benzoic acid at ~150°C for a couple of hours. This causes the Li^+ ions in a thin surface layer to be replaced by H^+ ions, resulting in an increased refractive index. The structure is then annealed at ~300°C for a few hours to diffuse the protons deeper into the crystal and form the low loss optical waveguide.

For this program we purchased and set up a temperature ~~stabilized~~ recirculating oil bath to heat the benzoic acid reservoir to a constant temperature. Annealing was performed in our high temperature, three zone furnace.

Channel waveguides were defined using photolithographic masking aligned to the poled domain grating. After fabrication and end face polishing, performed in house on a Logitech polishing machine, initial waveguides were characterized using a prism coupling technique to ensure that the mode effective refractive indices were as we expected. It is essential that we are able to predict these indices, at least to first order, so that we can predict the phasematching wavelength of the waveguide doubler.

7 Characterization of Waveguide Frequency Doublers

To characterize waveguide doubler devices we used a broadly tunable titanium sapphire laser pumped by an argon-ion laser. The infra-red output from the Ti:Sapphire was end coupled into the APE waveguide through a polished end face using a microscope objective, as indicated in figure 5. The generated blue light and transmitted IR were collected at the output of the waveguide by a second microscope objective, separated by a dichroic mirror, and monitored on calibrated silicon photodiode detectors. The wavelength of the Ti:Sapphire laser was monitored on a wavemeter or optical spectrum analyzer.

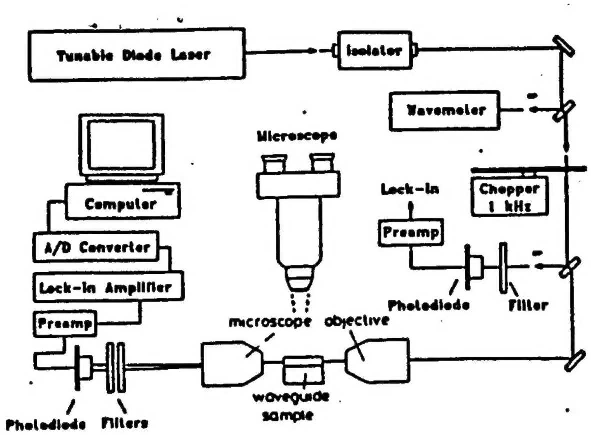

Figure 5 : Experimental set-up used to characterize waveguide frequency doublers

By scanning the wavelength of the Ti:Sapphire laser under computer control we measured the phasematching curves for a number of devices. Examples of these curves are displayed in figure 6. The phasematching curve for the (0,0) mode interaction is close to the ideal sinc^2 curve shape indicating high device quality. The device conversion efficiency of ~120%/W is roughly as expected from the number of merged patches in the domain grating on this particular device. The width of the central peak of the tuning curve indicates that the device has an effective phasematched length of 7.4mm, giving a normalized conversion efficiency of ~220%/W/cm^2 (the effective length is reduced slightly from the low power measurement of 7.6mm, probably due to heating effects). Note that this tuning curve was recorded at a generated blue power of 5mW, greater than 7mW of blue output has been generated, limited by the available pump power. The higher conversion efficiency displayed by the (0,1) mode interaction, ~300%/W with an interaction length of 9mm, indicates that our waveguide structure is not optimized in terms of refractive index and physical depth to achieve maximum conversion efficiency into the more desirable (0,0) mode.

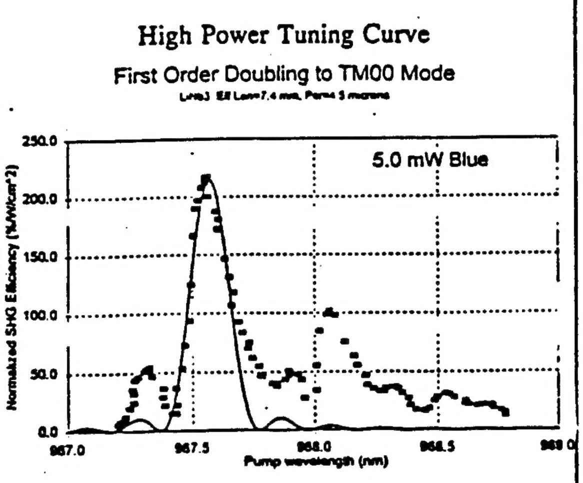

Figure 6a: Wavelength tuning curve of 1st order blue, waveguide SHG interaction [(0,0) mode]

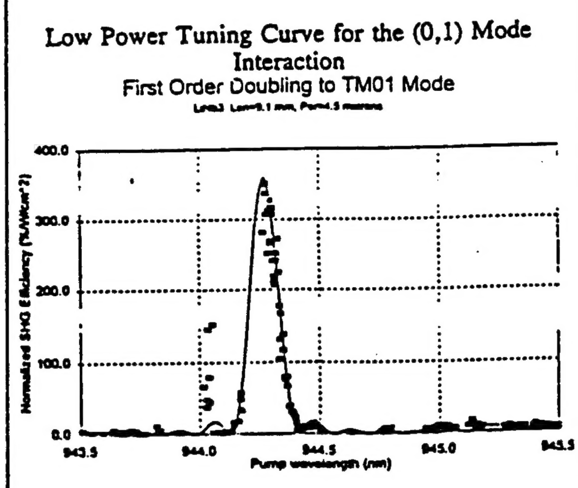

Figure 6b: Wavelength tuning curve of 1st order blue,. waveguide SHG interaction [(0,1) mode]

Figure 6 : Experimentally measured phasematching curves for $LiNbO_3$ waveguide frequency doublers